



False Black Pepper (*Embelia ribes*) Seeds-natural Colour Source for Wool Dyeing

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Authors' contributions

This work was carried out in collaboration between the authors. Author SK designed the study, wrote the protocol, managed the literature searches and wrote the manuscript. Authors MK actually conducted the experiment in the laboratory. Both authors read and approved the final manuscript.

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ABSTRACT

It is a need of the time to try out different sources of natural dyes to colour the textiles as the synthetic dyes used on textiles are carcinogenic and can create havoc in the human life system. The research was carried out for optimizing different conditions for dyeing wool with dye extracted from False Black Pepper seeds. The study was conducted in Department of Textiles and Apparel Designing, College of Community Science, Vasant Rao Naik Marathwada Krishi Vidyapeeth, Parbhani, Maharashtra (India). The dyeing conditions optimized for dyeing wool were, medium of dye extraction, dye extraction time, dye material concentration, dyeing time and mordant concentrations. The colour fastness of wool dyed by adopting optimized conditions were tested for different parameters. The alkaline medium was most suitable for extracting dye from False Black Pepper seeds for dyeing wool. On the basis of percent dye absorption the standardized dye extraction and dyeing time was 30 min, dye material concentration was 5%. The highest dye absorption percent was seen when wool was dyed with 10% Potassium Aluminum Sulphate (Alum) mordant in a simultaneous mordanting while for Copper Sulphate mordant 3% in post mordanting and for Ferrous Sulphate it was 3% in pre-mordanting. Wool dyed with False Black Pepper dye using different mordants adopting different mordanting methods exhibited very good to excellent

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colour fastness to sunlight, washing, rubbing and perspiration except wet rubbing as noticeable colour staining was observed in all the samples. The subtle, attractive colour range of light brown to dark brown acquired by the wool when dyed with False Black Pepper dye using different mordents.

Keywords: False black pepper dye; mordant; colour fastness; dyeing; wool.

1. INTRODUCTION

Since the last decade, the application of natural dyes on textile materials is gaining popularity all over the world [1]. Natural dyeing is gradually making its way in global market [2]. Natural dyes are dyes and pigments obtained from animal or herbal sources, acquired by no or minimal chemical treatments [3]. In dyeing with synthetic dyes enormous chemicals are released in environment in form of unused dye or industrial waste. [4] Globally, the textile dyeing industry is known to be a major contributor to environmental pollution [5,6]. Though synthetic dyes are widely available at economical price and produce a wide variety of colours; these dyes produce skin allergy, toxic wastes and other harmfulness to human body. In spite of the better performance of synthetic dyes, recently the use of natural dyes on textile materials has been attracting more and more scientists due to the wide availability of natural dyes and their huge potential. [7] The present scenario is focused more on the utilization of the vast diversity of natural resources of colour pigments for their use in food materials, pharmaceuticals and textiles, in place of their synthetic counterparts. [8] Presently increasing awareness about environment, pollution and ecology nationally and internationally has revived the adaptation of the old technique of natural dyeing leaving synthetic dyes behind in race. [9,10] The people are becoming more and more health-conscious and demands for producing textile products through environment- friendly and sustainable dyes and dyeing processes are increasing. Many carcinogenic and allergic synthetic dyes are banned now. [11,12] Vegetable sources of natural dyes are renewable. They are also non-toxic and non-allergic. They are biodegradable and eco-friendly [13]. Natural dyes have many excellent properties such as little side effect, high safety factor, biodegradable, green environmental protective. Some natural dyes have certain therapeutic effects and health functions [14]. Natural dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. [15] Natural dyes can produce special aesthetic qualities, which, combined with the ethical significance of a product that is environmentally friendly, gives

added value to textile production as craftwork and as an industry. [16] Natural dyes are used for small scale production, so less capital is required for establishing natural dyeing plant. Natural dyeing may help to lower poverty from the society.

Embelia Ribes also known as False Black Pepper, is a large woody tropical forest shrub with slender branches and dotted leaves. Stems are whitish- gray and roots are brownish-gray with hairy reddish roots. Fruits are small, smooth and succulent which encompasses reddish seeds that run slightly black. The berries are considered to be carminative, digestive, laxative and useful for treating pneumonia and dropsy. Dried fruits are used to prepare medicines and used as anti-carminative, anti-helminthic, anti-inflammatory, anti-bacterial, anti-astringent and anti-diuretic. [17] The research was carried out for optimizing different conditions for dyeing wool with dye extracted from False Black Pepper seeds.

2. METHODS

2.1 Dye Source

False Black Pepper seeds were a dye source for this study. The first step in creating a natural dye for wool or whatever you hope to add colour to, is to gather the plant material. [18] False Black Pepper seeds can be collected from plants. For the experiment the seeds were procured from the market, cleaned well and made into powder.



Fig. 1. False black pepper seeds

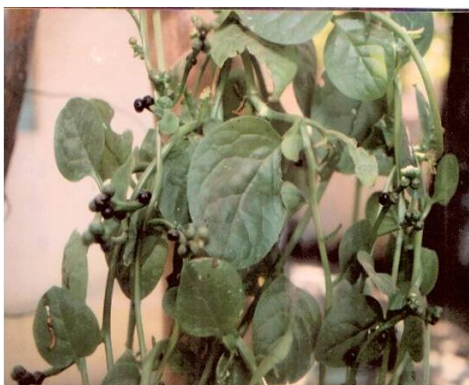


Fig. 2. Plant of false black pepper

2.2 Preparation of Wool for Dyeing

Before dyeing wool yarns were scoured to remove all impurities. The scouring solution was prepared by adding 2 gm of a neutral soap in one litre of water. The scouring was done for 30 min. with 1:50 material to liquor ratio. After scouring the yarns were washed many times to remove traces of soap and then were dried.

2.3 Medium of Dye Extraction

Three media i.e. aqueous, alkaline and acidic were tried out for extracting colour from False Black Pepper seeds. For aqueous media distilled water was used while an acidic solution was prepared by dissolving 1 ml of acetic acid in 100 ml of water and the alkaline solution was prepared by dissolving 0.5 gm of sodium carbonate in 100 ml of water separately. One gram of powdered dye material was added in 100 ml of each solution and boiled for 30 min. One gram wool was added in each medium and dyeing was done for 30 min. in all the solutions. The optical density (O.D) of dye solution before and after dyeing was recorded and percent dye absorption was calculated by using formulae.

$$\text{Percent dye absorption} = \frac{\text{O.D. before dyeing} - \text{O.D. after dyeing}}{\text{O.D. before dyeing}} \times 100$$

2.4 Optimization of Dye Extraction Time

One gram dye material was boiled in 100 ml of alkaline solution each for 30 min., 45 min. and 60 min. respectively. Dyeing of one gram of wool yarn was carried out for 30 min. in each extract. On the basis of percent dye absorption value the dye extraction time was optimised.

2.5 Optimization of the Concentration of Dye Material

Dye solutions of varying concentrations from 1 gm to 10 gm of False Black Pepper powder per 100 ml of alkaline solution were prepared separately and one gram of yarn was dyed in each solution for 30 min. The optical density of each solution before and after dyeing was recorded and percent dye absorption value was calculated. The optimum concentration of False Black Pepper seed powder was determined based on maximum dye absorption.

2.6 Optimization of Dyeing Time

Dyeing was carried out in dye solutions prepared using optimum dye material concentration for 30, 45 and 60 min separately for optimizing dyeing time. The optical density of each solution before and after dyeing was recorded and dye absorption percent at the varying time was calculated. Optimum dyeing time was determined based on maximum dye absorption.

2.7 Optimization of Mordant Concentration

Natural dyes generally require mordant. [19] Mordents used for the study were

Potassium Aluminum Sulphate (Alum)	5% to 20%
Copper Sulphate	1% to 5%
Ferrous Sulphate	1% to 5%

With these mordents wool was mordanted in three mordanting methods i.e. pre- mordanting, simultaneous mordanting and post mordanting. In pre mordanting wool was mordanted before dyeing. In simultaneous mordanting method dyeing and mordanting were carried out at the same time in the same dye bath and in post mordanting yarns were dyed first and then mordanted. The optimum concentration of each mordant for each mordanting method was determined on the basis of percent dye absorption.

2.8 Dyeing

The dye extracted in an alkaline solution using 5% concentration of False Black Pepper seed powder was neutralised first and then the scoured wool was dyed in extracted dye adopting all the optimised conditions of dyeing and mordanting.

2.9 Colour Fastness Tests

Wool dyed with False Black Pepper dye was tested for fastness to sunlight, washing, rubbing and perspiration. The fastness test of sunlight was carried out according to ISI:686-1985 test. The washing fastness test was as per ISI test no. 2 (IS 3361-1979). Fastness against rubbing was determined by using IS: 766-1988 test and fastness for perspiration was evaluated adopting IS:971-1983 test. The dyed wool was graded for sunlight fastness by using Blue Wool Standard and graded for colour change & colour staining in washing, rubbing and perspiration by using Gray Scales.

Images of Dyed Wool

Wool dyed with False Black Pepper dye using different mordants



Fig. 3(a). Potassium aluminum sulphate (Alum)



Fig. 3(b). Copper sulphate



Fig. 3(c). Ferrous sulphate

3. RESULTS AND DISCUSSION

Table 1 shows optimized conditions for dyeing wool with False Black Pepper dye. Based on percent dye absorption, the alkaline medium was found to be most suitable for the extraction of dye. Other optimized conditions for dyeing wool with False Black Pepper dye were dye material concentration 5%, dye extraction as well as dyeing time 30 min. Optimised concentration of Alum mordant for pre-mordanting, simultaneous mordanting and post mordanting were 10%, 10% and 5% respectively and for Copper Sulphate mordant for pre-mordanting 1%, simultaneous and post mordanting 3% each while for Ferrous Sulphate mordant pre-mordanting 3%, simultaneous mordanting 2 post mordanting 1%.

Table 2 shows fastness grades of Alum mordanted & False Black Pepper dyed wool. Based on percent dye absorption value the optimized concentrations of Alum mordant for pre-mordanting, simultaneous mordanting and post mordanting were 10%, 10% and 5% respectively. The pre and post mordanted samples exhibited excellent to outstanding and simultaneous mordanted samples showed outstanding sunlight fastness. When subjected to washing tests, slight to noticeable colour change was seen in pre-mordanted, slight colour change in simultaneous mordanted and negligible to slight in post mordanted samples. Irrespective of mordanting method no colour staining was observed to washing. No colour change was observed in dry & wet rubbing but slight to noticeable colour staining was found in wet rubbing. Colour change was negligible to slight in pre and simultaneous mordanted and negligible in post mordanted wool in acidic

Table 1. Optimized conditions for dyeing wool with false black pepper dye

Medium of dye extraction	Dye material Conc.	Dye extraction time	Dyeing time	Mordanting		
				Mordant used	Mordanting methods	Mordant Conc. %
Alkaline	5 %	30 min	30 min	Alum	Pre	10
					Simultaneous	10
					Post	5
				Copper Sulphate	Pre	1
					Simultaneous	3
					Post	3
				Ferrous Sulphate	Pre	3
					Simultaneous	2
					Post	1

Table 2. Fastness grades of wool dyed with false black pepper dye using Alum mordant

Mordanting method	Mordant Conc. gm/100 gm wool	Dye Absorption %	Sun light	Fastness properties												
				Washing			Dry rubbing		Wet rubbing		Acidic perspiration			Alkaline perspiration		
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	10	59.25	7/8	3/4	5	5	5	3/4	5	3/4	4/5	4/5	4/5	4	5	5
Simultaneous Mordanting	10	81.67	8	4	5	5	2/3	3/4	5	3/4	4/5	5	5	4/5	4/5	4/5
Post Mordanting	5	64.86	7/8	4/5	5	5	5	3/4	5	3	5	5	5	5	5	5

Dye Percentage: 5%; Dyeing time: 30 min; Dye extraction time: 30 min; Extraction medium: Alkaline; Mordanting time: 30 min

Note : CC : Colour Change; CS : Colour Staining; C : Cotton; S : Silk

Table 3. Fastness grades of the wool dyed with false black pepper dye using copper sulphate mordant

Mordanting method	Mordant Conc. gm/100 gm wool	Dye absorption %	Sun-light	Fastness properties												
				Washing			Dry rubbing		Wet rubbing		Acidic perspiration		Alkaline perspiration			
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	1	56.36	7	4	5	5	5	4	5	2/3	5	5	5	5	5	
Simultaneous Mordanting	3	67.56	7/8	4/5	5	5	5	3/4	5	3/4	5	5	5	4/5	5	5
Post mordanting	3	69.06	7/8	3/4	5	5	5	3/4	5	3/4	5	5	5	4/5	5	5

Dye Percentage: 5%; Dyeing time: 30 min; Dye extraction time: 30 min; Extraction medium: Alkaline; Mordanting time: 30 min; CC : Colour Change; CS : Colour Staining; C : Cotton; S : Silk

Table 4. Fastness grades of the wool dyed with false black pepper dye using ferrous sulphate mordant

Mordanting method	Mordant Conc. gm/100 gm Wool	Dye absorption %	Sun light	Fastness properties												
				Washing			Dry rubbing		Wet rubbing		Acidic perspiration		Alkaline perspiration			
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	3	76.04	8	4	5	5	5	3	5	3	4/5	5	5	4/5	5	5
Simultaneous Mordanting	2	75.24	7/8	3/4	5	5	5	3/4	5	3	4/5	5	5	4/5	5	5
Post Mordanting	1	59.57	7/8	4/5	5	5	5	4	5	3/4	*	5	5	4/5	5	5

*Dye Percentage: 5%; Dyeing time: 30 min; Dye extraction time: 30 min; Extraction medium: Alkaline; Mordanting time: 30 min; CC : Colour Change; CS : Colour Staining; * : Hue change; C : Cotton fabric; S : Silk fabric*

perspiration test while in alkaline perspiration test pre mordanted samples showed slight colour change, simultaneous mordanted samples exhibited negligible to slight and post mordanted exhibited negligible colour change. No colour staining was noticed in both acidic & alkaline perspiration tests except negligible to slight colour staining in pre mordanted samples in acidic perspiration and in simultaneous mordanted samples in alkaline perspiration test.

Fastness grades of False Black Pepper dyed and Copper Sulphate mordanted wool are given in Table 3. The optimized concentration of Copper Sulphate mordant for pre-mordanting was 1% while for simultaneous mordanting and post mordanting it was 3%. Excellent to outstanding sunlight fastness was observed in all the samples. Negligible to slight change in colour in pre & simultaneous mordanted, slight to noticeable in post mordanted and no colour staining was found in tested samples in washing. No colour change while slight to noticeable colour staining was observed in both dry rubbing and wet rubbing tests. No colour change or staining was seen in acidic perspiration test but simultaneous and post mordanted samples showed negligible colour change in alkaline perspiration test.

Fastness grades of Ferrous Sulphate mordanted and False Black Pepper dyed wool are tabulated in Table 4. Three percent was the optimized concentration of Ferrous Sulphate mordant for pre-mordanting followed by simultaneous mordanting 2% and for post mordanting it was 1%. Sunlight fastness found to be outstanding in pre mordanted and excellent to outstanding in simultaneous and post mordanted samples. In washing tests slight colour change in pre-mordanted, slight to noticeable in simultaneous mordanted and negligible to slight colour change in post mordanted samples were seen but no colour staining was observed in the tested samples. There was no colour change but slight to noticeable colour staining in both dry and wet rubbing. In acidic as well as alkaline perspiration tests dyed wool found to have negligible to slight colour change but the post mordanted samples with 1% mordant exhibited hue change in acidic perspiration test. Dyed wool was found to be resistant to colour staining in acidic and alkaline perspiration tests.

4. CONCLUSION

It is concluded that the alkaline medium was most suitable for extracting dye from False Black

Pepper seeds for dyeing wool. On the basis of percent dye absorption the standardized dye extraction and dyeing time was 30 min. The optimized dye material concentration for dyeing wool was 5%. The highest dye absorption percent with Potassium Aluminum Sulphate (Alum) mordant was 10% in simultaneous mordanting while for Copper Sulphate it was 3% in post mordanting and for Ferrous Sulphate it was 3% in pre-mordanting. Wool dyed with False Black Pepper dye using different mordents adopting different mordanting methods exhibited very good to excellent colour fastness to sun light, washing, rubbing and perspiration except wet rubbing as noticeable colour staining was seen in wet rubbing tests in all the samples mordanted with different mordents and mordanting methods.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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